A STUDY OF THE EFFECTS OF VARIATIONS OF CONCENTRATION AND SURFACE TREATMENT ON ADSORPTION IN A METALLIC COPPER-IODIDE ION SYSTEM

GEORGE ANTHONY LYNN

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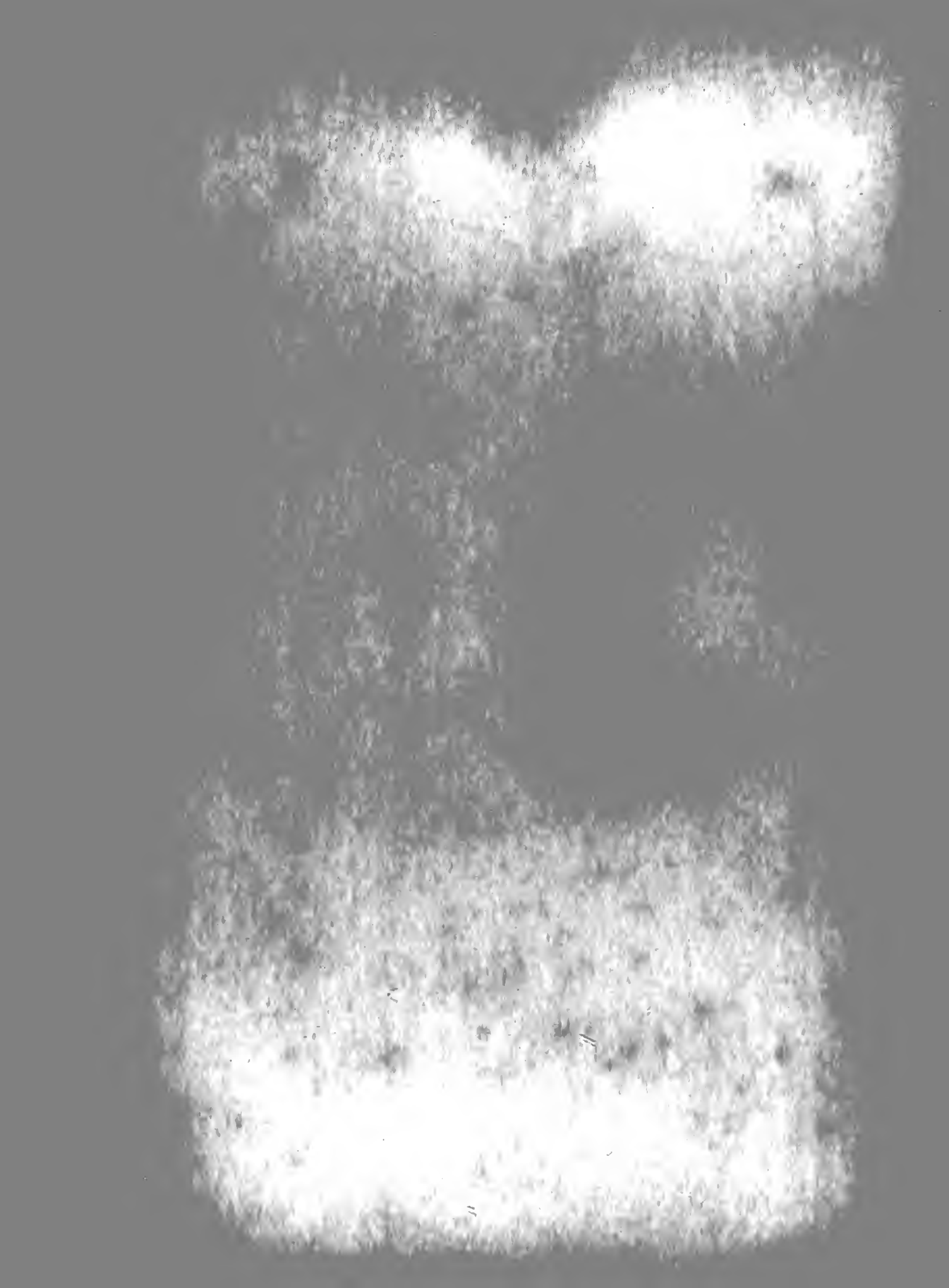
George A. Lynn



A STUDY OF THE EFFECT OF VARIATIONS OF CONCENTRATION AND SURFACE TREATMENT ON ADSORPTION IN A METALLIC COPPER-IODIDE ION SYSTEM

by

George Anthony Lynn

Captain, United States Army

Submitted in partial fulfillment
of the requirements
for the degree of
MASTER OF SCIENCE
IN
PHYSICS

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Monterey, California

1 9 5 4

This work is accepted as fulfilling

the thesis requirements for the degree of

MASTER OF SCIENCE

IN

PHYSICS

from the

United States Naval Postgraduate School

PREFACE

This study was conducted at the United States Naval Postgraduate School in Monterey, California, during the spring of 1954 in partial fulfillment of the requirements for the degree of Master of Science in Physics.

The author wishes to express his appreciation to Professors Gilbert F. Kinney and William W. Hawes for their assistance, cooperation, and guidance in the preparation of this paper.

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INTRODUCTION

Part I

Adsorption has been defined[1] as the tendency of a solid to condense upon its surface a layer of any gas or solute with which it may be in contact. The amount of material adsorbed may vary depending upon such factors as temperature, concentration or pressure of the adsorptive, condition of the surface of the adsorbent, and the physical and chemical properties of the adsorbent and the adsorptive. A more general definition[2] states that a substance is adsorbed if the concentration of the substance in a boundary region is higher than in the interior of the system. The definition of adsorption rests on measurements of concentration, and carries no implication of mechanism.

Adsorption takes place at the interface between adjacent phases, and the interface may be solid-liquid, solid-gas (or vapor), liquid-liquid, or liquid-gas (or vapor). In a reversible process, the molecules can return to the phase from which they came, their place being taken by other molecules; and at equilibrium, the rate of departure equals the rate of arrival of the molecules at the interface.

All adsorptive processes are exothermic, and the decrease in the heat content of the system is called the heat of adsorption. Adsorption at solid surfaces may be either physical (van der Waals) or chemical, according to the magnitude of the energy change involved. There is no rigid distinction, however, as adsorptive processes may involve

1. F. H. Getman and F. Daniels, Outlines of Physical Chemistry. John Wiley and Sons, New York, 1940, p 255.
2. H. G. Cassidy, Adsorption and Chromatography. Interscience Press, New York, 1951, p 1.

energy changes ranging from relatively small in physical adsorption to the order of magnitude of those found in chemical reactions in chemical adsorption. In intermediate ranges, the process may be thought of as involving both physical and chemical adsorption simultaneously, although this has not been clearly shown.

A third classification of adsorption, activated adsorption, has been described by Adam.[3] Activated adsorption takes place slowly and at about room temperature. Here it is postulated that in order for adsorption to take place, some sort of potential barrier must be overcome by the molecules, so that only those with sufficient energy can be adsorbed. At room temperature, the energy distribution may be such that only a relatively few molecules possess sufficient energy to surmount the barrier, and in such situations an increase in temperature would increase the rate of adsorption.

When adsorption occurs from a mixture of substances, as from a solution, there will be considerable competition for the interface. It is here that the geometry of the surface and the molecules of the adsorptive are of great importance, as well as the physical and chemical properties of the substances.

The adsorbent introduces many factors which might have an influence on the adsorptive process. The physical composition of the surface will certainly have an effect on adsorption.[4] Langmuir has shown that on a plane, crystalline, chemically homogeneous surface the disposal of sites on which adsorption can take place will be in a regular pattern. Any irregularities in the pattern lead to the formation of so-called "active

3. N. K. Adam, The Physics and Chemistry of Surfaces, 3rd Ed. Oxford University Press, London, 1941, p 252.
4. Cassidy, op. cit., p 43 ff.

centers". On an amorphous surface regularity may or may not exist. The spaces available on a plane crystal may be of several kinds, but the regularity of the surface may lead to some stoichiometric relation in the adsorption of different substances. At the edges and corners of an adsorbent certain molecules will be in a somewhat more exposed position. Such molecules might be more active in adsorption than others.

If the surface is rough, then more of the surface molecules will be in an exposed position, and the roughness might therefore be more conducive to increased adsorption of small molecules per unit number of surface molecules over the plane surface, while with large molecules the effect might be opposite. If the surface contains capillaries and fissures, adsorption may be favored for those small molecules which can enter these imperfections in the surface, while larger molecules may therefore have large areas of the surface excluded to them for the purpose of adsorption.

Smooth surfaces are extremely difficult to obtain. Frazer, Patrick, and H. E. Smith[5] observed that on glass surfaces which had never been in contact with liquids, the adsorbed layer was probably one molecule thick, whereas on glass that had been etched by contact with liquids the adsorbed layer was probably several molecules thick. It is possible that some surface imperfections can have a negligible effect, since at the interface mutual interaction between adsorbed molecules may cause them to adhere laterally and so influence the quantity adsorbed by allowing the adsorbed film to bridge roughness and fissures in the adsorbent.

5. Frazer, Patrick, and H. E. Smith, J Phys Chem, 31, 897 (1927).

Part II

In the case with which this study is concerned, that of a solid-solution type of interface, there are several features which must be considered. The liquid is homogeneous, due to the thermal motions of its particles. The solid surface, on the other hand, is very likely to be non-homogeneous, resulting in effects which will be discussed more fully later in this paper. We also have to consider that both the solvent and the solute molecules will have a tendency to be adsorbed, the effect here being dependent upon the nature of the solvent and the solute, with particular reference to the possibility of interaction, dissociation, or incompatibility. Since in this study extremely low concentrations of solute are employed, errors due to adsorption of the solvent are expected to be negligible, since there is a tendency toward preferential adsorption of the component present in the smaller concentration. Furthermore, it is not expected that effects due to interaction, dissociation, or incompatibility will affect the qualitative significance of the results.

In general, the recent literature is relatively sparse insofar as work on the rate of adsorption at solid-solution interfaces is concerned. There has been a considerable amount of work reported on gas adsorption on solids, and it is to be expected that this process should be in some way related to adsorption from solutions. An example of this work is the report by Russell and Stokes[6] of their study of the adsorption of hydrogen on alumina. They report a very high initial adsorption rate followed by a sharp drop to lower rates. In work with solutions,

6. A. S. Russell and J. J. Stokes, Jr., J. Am Chem Soc, 69, 1316 (1947).

Zettlemoyer and co-workers[7] have reported on the adsorption of fluoride from aqueous solutions, their results similarly showing a high initial rate followed by a sharp change to lower rates. Some recent work employing radioactive isotopes has been reported by King and Schochet.[8] They studied the adsorption of silver salts on silver using Ag^{110} as a tracer, and their findings followed the pattern described above. In addition, they reported that treating the silver with dilute nitric acid prior to use resulted in an increase in the amount of Ag^{110} adsorbed. Rydberg and Rydberg[9] have reported on the adsorption on glass and polyethylene of Th^{232}, present in solution in tracer concentrations. They found a very high initial rate of adsorption (half time 15-25 minutes) and a very slow rate after about one hour (half time 7 hours). They also noted a sharp adsorption maximum on glass around pH 4.2.

The purpose of this study is twofold. First, it is to observe the effect of changes in the concentration of the solution on the rate of adsorption and on the total amount of substance adsorbed, and to observe the effects of surface imperfections on the overall patern of adsorption. The second is to lay the groundwork for future research in this field at this institution by devising basic techniques which can be adapted to the many phases of this particular field.

In this study, radioactive iodine in very low (tracer) concentrations was the adsorptive, and metallic copper in the form of thin disks was the adsorbent. Results indicate that in general, the overall

7. A. C. Zettlemoyer, E. A. Zettlemoyer, and W. C. Walker, Jr., J Am Chem Soc, 69, 1312 (1947).
8. King and Schochet, J Phys Chem, 57, 895-8 (1953).
9. Rydberg and Rydberg, Svensk Kem Tidskr, 64, 200-11 (1952).

adsorption process can be broken down into two steps or phases whose combined effect is observed. Surface imperfections and variations in preparatory treatment exert a strong influence on the adsorption processes, and may be capable of overriding concentration effects. The total amount of iodide adsorbed varied directly with concentration, except in those cases where surface effects appeared to exert severe inhibitions on the adsorption process. The rate of adsorption was initially high, and fell rapidly to low values within a few hours at most, following closely the pattern observed by others.

Experimental Procedure

Many experiments on adsorption employ the technique of measuring the concentration of the adsorptive before and after the adsorbent has been permitted to perform its function, with the difference being considered adsorbed. With the current availability of numerous radioactive isotopes, a more direct method of studying adsorption phenomena is provided. It is necessary to make one basic assumption when using radioactive isotopes in the study of adsorption: it is assumed that an active molecule or atom will act in the same manner as an ordinary molecule or atom. Insofar as the phenomenon of adsorption is concerned, the writer feels that this assumption is indeed a valid one. Certainly the active isotope will react chemically in the same manner as an isotope which is not active, and while it may possess additional energy which can be manifested in the release of some form of radiation, this energy does not normally affect its physical properties.

Using this assumption as a basis, we further postulate that the amount of activity on a solid adsorbent which has been immersed in an active solution will be proportional to the amount of adsorptive present. By immersing the adsorbent for varying periods of time, and measuring its activity between immersions, a picture of the activity (and consequently the amount adsorbed) as a function of time is presented. From such a picture, it is relatively simple to differentiate the curve and so determine the rate of adsorption as a function of time.

In deciding on the isotope to be used in this study, consideration was given to several desirable characteristics. First, the isotope should be readily available and comparatively inexpensive. The reasons for this are obvious. Second, the isotope should have a fairly short half-life--

perhaps no longer than two or three weeks, but no shorter than two or three days. The upper limit on the length of half-life comes about from the viewpoint of laboratory contamination, in that an accident with such an isotope will not prevent the use of the laboratory for long periods of time. This is especially important where the facilities must be available for several groups concurrently. Use of shorter half-life materials than the lower limit proposed will result in too rapid decay of the solution, which would be somewhat of a handicap in long test runs. Third, in order to avoid the necessity for elaborate precautions in handling the active solutions and solid adsorbents, it would seem advisable to choose an isotope with relatively low energy radiation, with beta radiation preferable to gamma.

Based on the above considerations, the isotope I^{131} was chosen. It has an 8-day half-life and emits beta radiation of about 0.6 Mev.

As the solid adsorbent, metallic copper was chosen since it is readily available in a relatively pure state. Further, it etches easily, thus making it easier to obtain uniformity of surface without elaborate pre-treatment. The copper used in this study was prepared in the form of thin disks or planchets of a convenient size.

The actual technique of immersing the copper planchets into the aqueous solution of iodide may be described as follows. A small hole was drilled in each planchet, and a piece of copper wire hooked into it. This provided an easy means of removing the planchet after it had been immersed for the desired time. The planchet was then rinsed lightly to remove any droplets of solution, after which the planchet was dried with a tissue and placed in a holder for counting. Tests were made to

ascertain whether or not the activity on the reverse side would affect the count from the side facing the tube. It was found that the thickness of the planchet was sufficient to shield out completely the radiation from the under side, thus eliminating this as a factor. It should be mentioned here that since any given test run took place over an extended period of time--in some cases as much as a week--it was necessary to correct all counts for decay back to the starting date in order to affect a normalization of the data. By so correcting the data which might be obtained a few days after the initial readings, the atoms or molecules which had been adsorbed and then undergone decay before a count could be taken were effectively included. This obviously must be done, or a considerable amount of adsorptive will go undetected.

The activity of the planchets was determined with the customary setup of a Geiger-Mueller tube in conjunction with an automatic scaler. The tube employed was a Tracerlab TGC-1 type end-window counter. The scaler was a CP-37UD Radiac Computer-Indicator also manufactured by Tracerlab. The radioactive iodine was purchased as needed from Tracerlab. The tube was carefully calibrated to determine its characteristics and efficiency factors, so that when determining the concentration of the solutions it could be done on an absolute basis as of the time of measurement. Geometry was fixed by means of a standard holder, which provided a choice of several counting positions.

The surface of the planchets was examined under a low-power microscope, and in general the surface was found to contain many imperfections. These included minute cracks and fissures, as well as small pits and gouges. Many of these were not visible to the unaided eye.

In addition, the surfaces were often dirty. In order to clean up the surfaces and attempt to minimize the effect of the imperfections, the planchets were treated for three minutes in a bath of dilute nitric acid, after which they were rinsed in water to remove excess acid. In one case, the planchets were so prepared and then stored under water for five days prior to use. The effect of this treatment, as opposed to the usual treatment of immediate use after preparation, is discussed with other experimental results. The action of the acid bath removed surface dirt and had some effect in smoothing out pits and gouges. The fissures and cracks were not noticeably affected, insofar as visual examination could determine.

In order to determine if excessive activity losses were occurring during the course of a test run, the concentration of the solution was measured before and after a test run, the latter figure being corrected for decay. In all cases, a small amount of activity was lost, and in accounting for the loss part of it could be traced to the amount of active material adsorbed as measured by the activity on the planchets. The rest of the loss was presumed to have occurred in the process of rinsing solution droplets off the planchets prior to taking a count, and also in adsorption into the glass surfaces of the beaker, stirring rod, and thermometer. In no case was the entire amount lost excessive.

Experimental Results

The total adsorption pattern was normal. It was expected that the total amount of iodide adsorbed would be greater at the higher concentrations than at the lower ones, but that the percent adsorbed would be greatest at the lowest concentration. Table I illustrates these results, with the figures for total adsorption representing one side of the planchet only. This gives a qualitative picture, which is sufficient for the purpose of this study.

Table I
Adsorption after 27 hours

Concentration	Total Activity	Total Adsorbed	% Adsorbed
.283 μc/ml (300 ml)	84.9 μc	0.88 μc	1.04%
.096 μc/ml (500 ml)	48.0 μc	0.58 μc	1.21%
.0325 μc/ml (225 ml)	7.31 μc	0.116 μc	1.59%

Not included in this table are the figures for test runs 4 and 5, since in these runs the amount of activity present on the planchets was low due to surface effects. These surface effects will be discussed later in this section.

The log activity-1/t plots of the test runs are presented in two groups, of which the second will be discussed under surface effects. The first group, including runs 1,2,3, and 6, shows the general picture of the adsorption isotherm, which can be represented by an equation of the form $\alpha = Ae^{-a/t} + Be^{-b/t}$. The particular equations calculated for each of these runs are as follows:

$\alpha = 1700\ e^{-.102/t} + 18000\ e^{-2.16/t}$ (.283 μc/ml concentration)

$\alpha = 3900\ e^{-.172/t} + 7400\ e^{-1.05/t}$ (.096 μc/ml concentration)

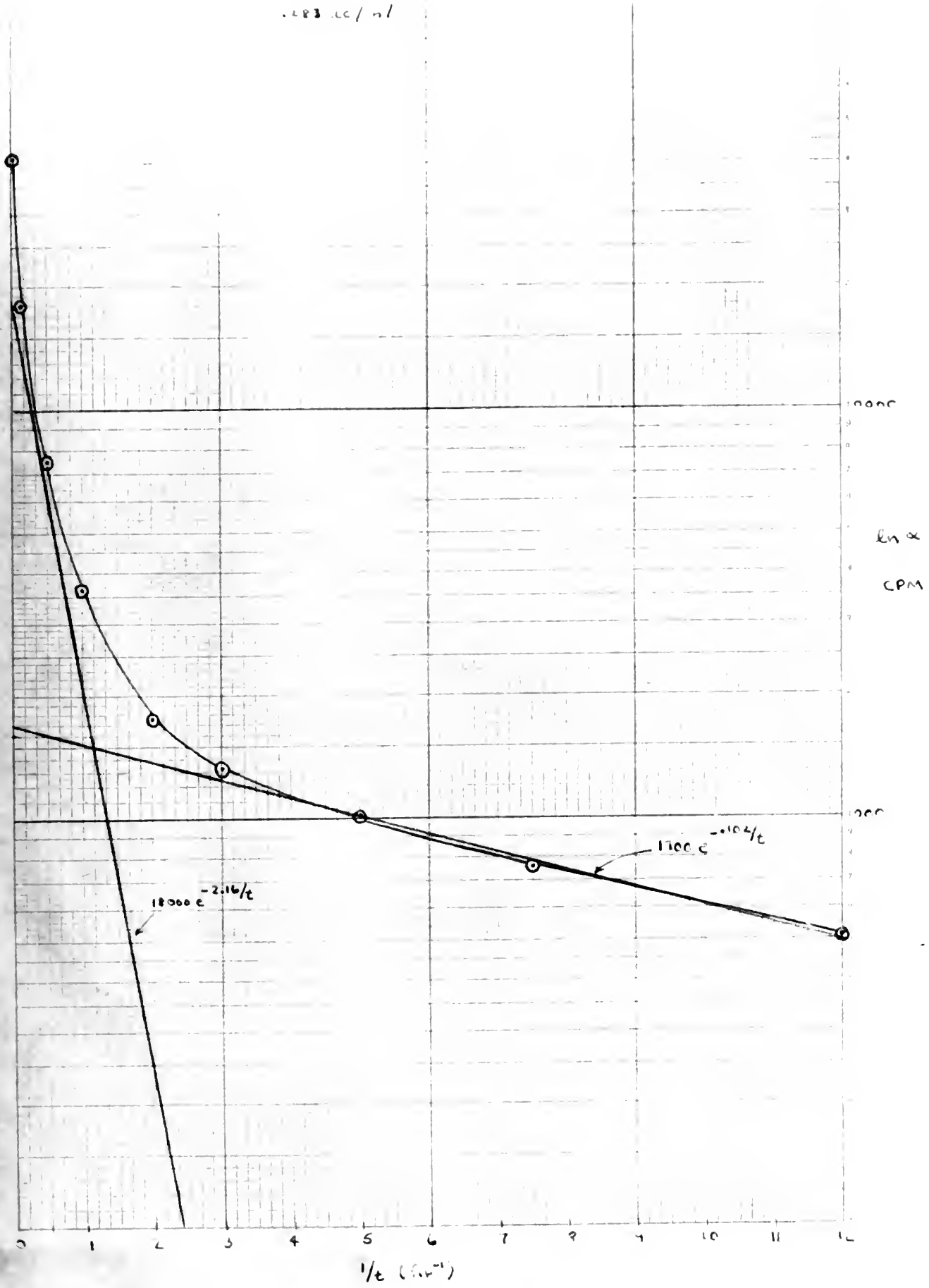
$\alpha = 1100\ e^{-.070/t} + 1500\ e^{-2.18/t}$ (.088 μc/ml concentration)

$\alpha = 850\ e^{-.075/t} + 2200\ e^{-2.08/t}$ (.0325 μc/ml concentration)

I-1

Ln α - 1/t

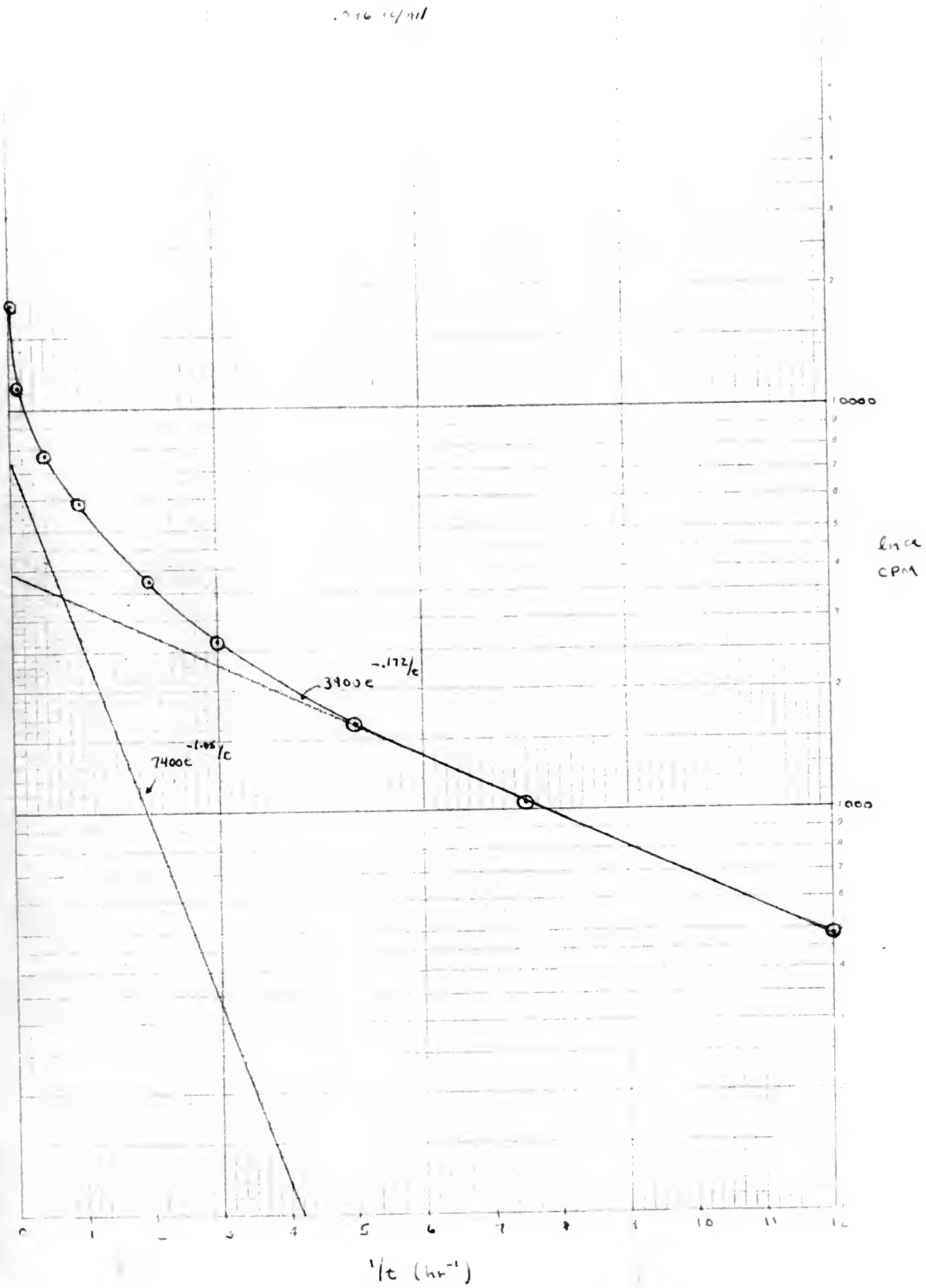
.283 cc/ml

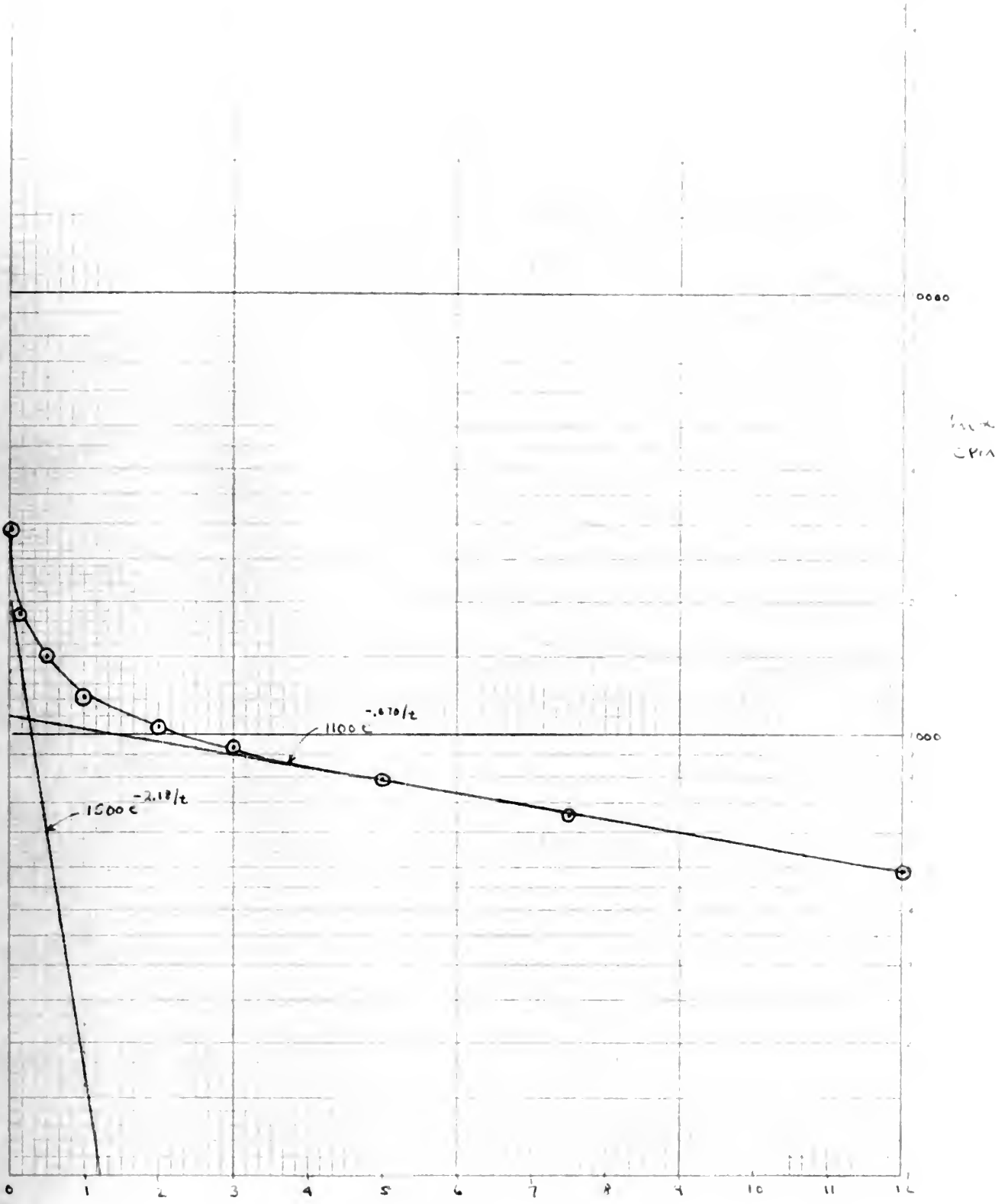


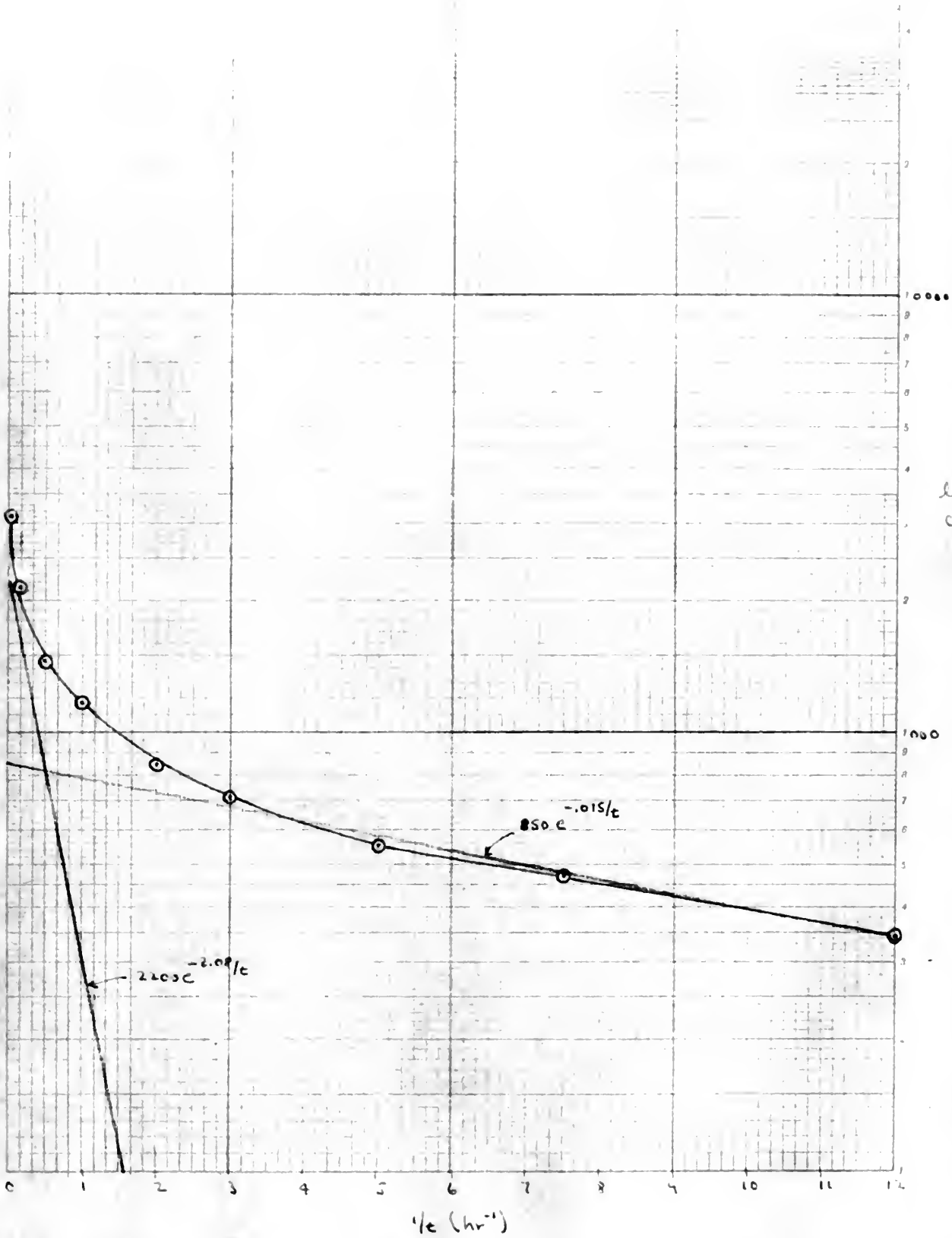
I-2

- 1/t

.176 cpm/ml




0000
000
1100 e^-.670/t
1500 e^-2.18/t
0
1
2
3
4
5
6
7
8
9
10
11
12
1/t (hr^-1)


10000
1000
ln α
CPM
850 e^-.015/t
2203 e^-2.08/t
0
1
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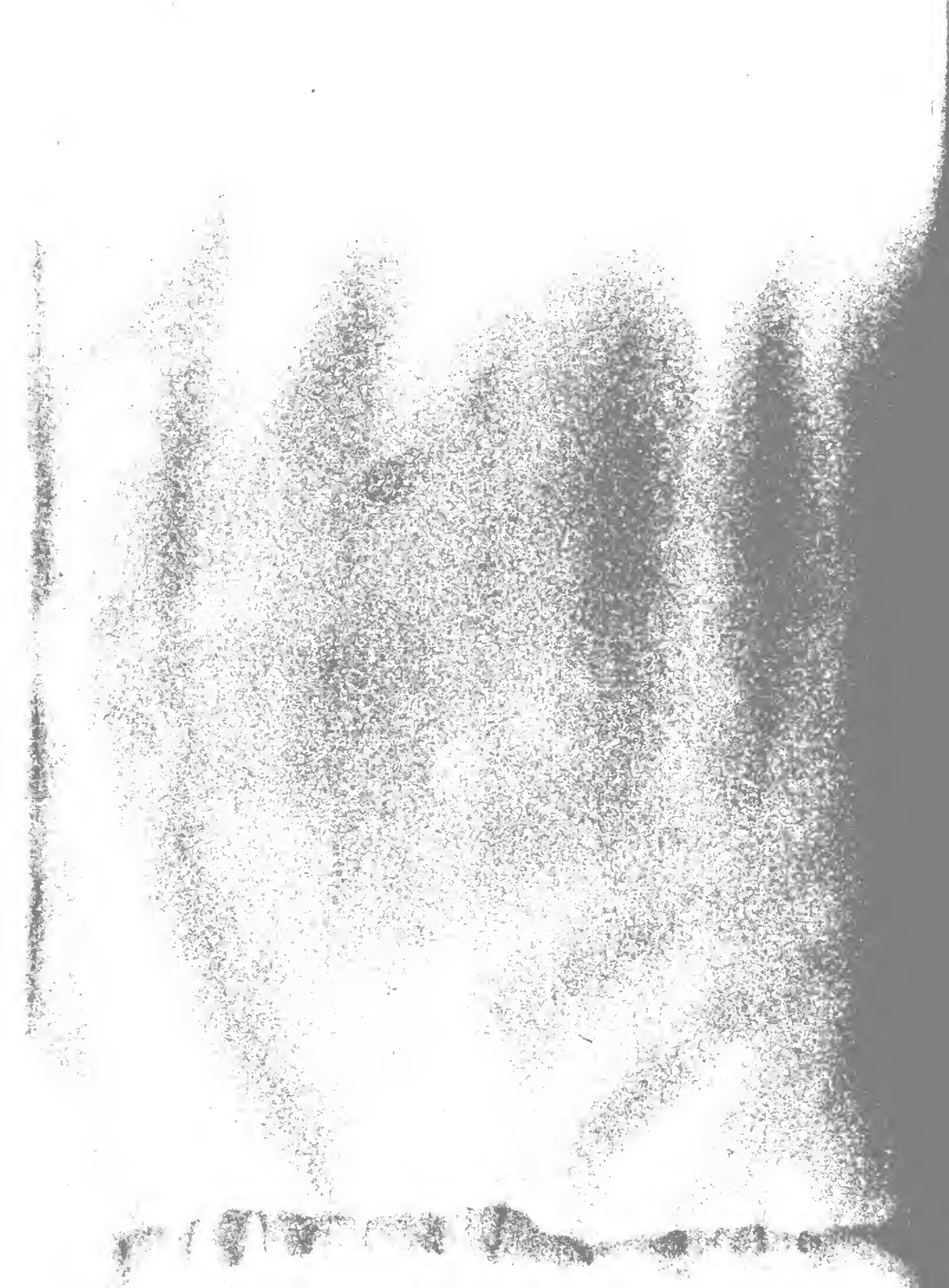
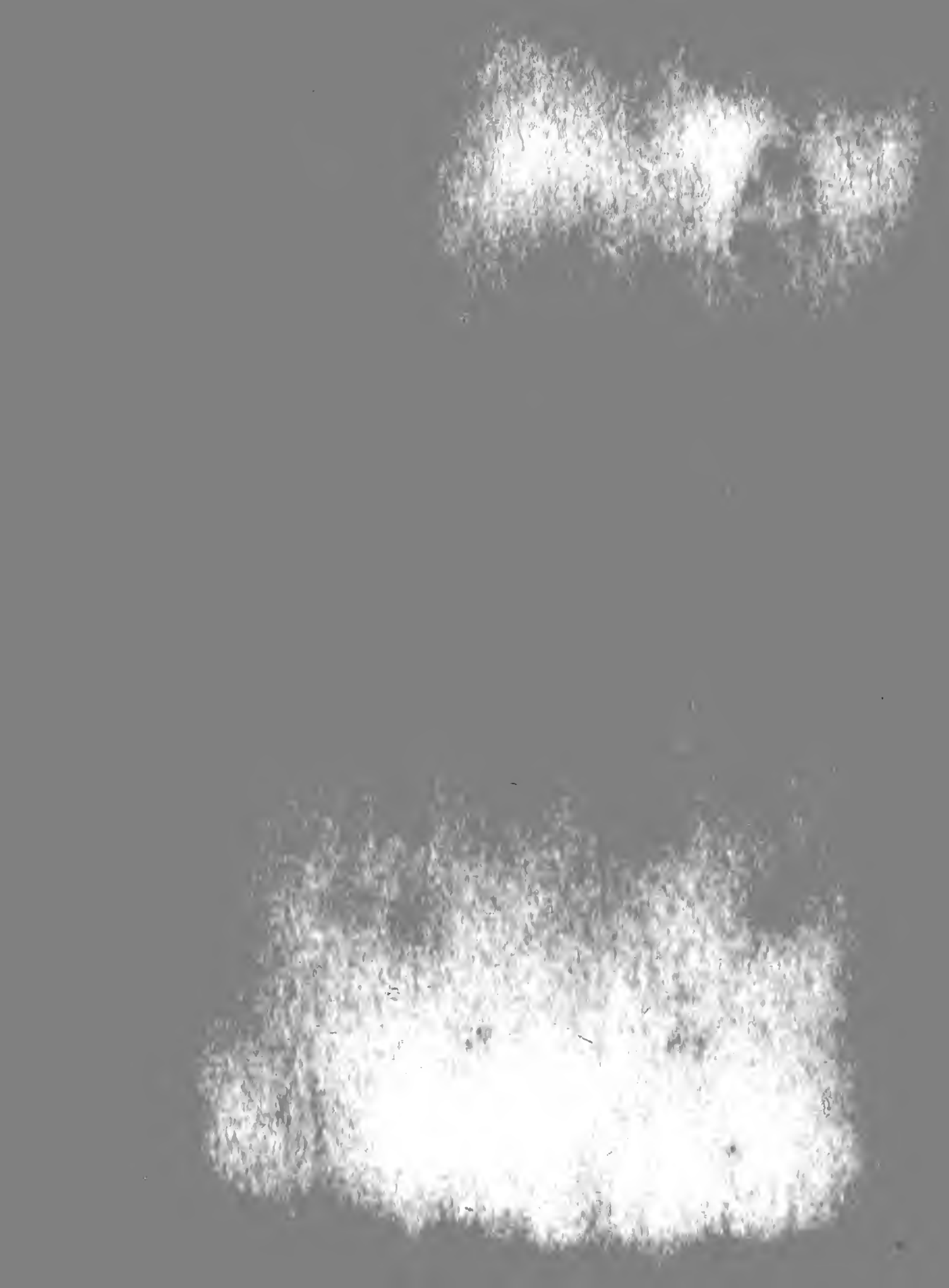
11
12
1/t (hr^-1)



A study of these relations indicates little correspondence between the changes in the various constants and the changes in concentration. This lack of correspondence indicates that concentration effects may easily be obscured by the action of surface imperfections. These results may also indicate that control of concentration was not as precise as expected. This is possible since the amount of substances, other than radioactive iodine, which may have been present in the active material as purchased could easily vary from lot to lot, both as to type and amount of material.

As a possible explanation of the lack of correspondence, we may postulate that the linear portion of the curve, which extends on the average for 12 to 20 minutes, represents a particular adsorption process which is more sensitive to variations in surface than it is to concentration changes. The second portion of the curve is believed to indicate a second process which initially is negligible in extent. This process might be more readily influenced by concentration changes, although it would also be affected by the initial process which had been and was still going on.

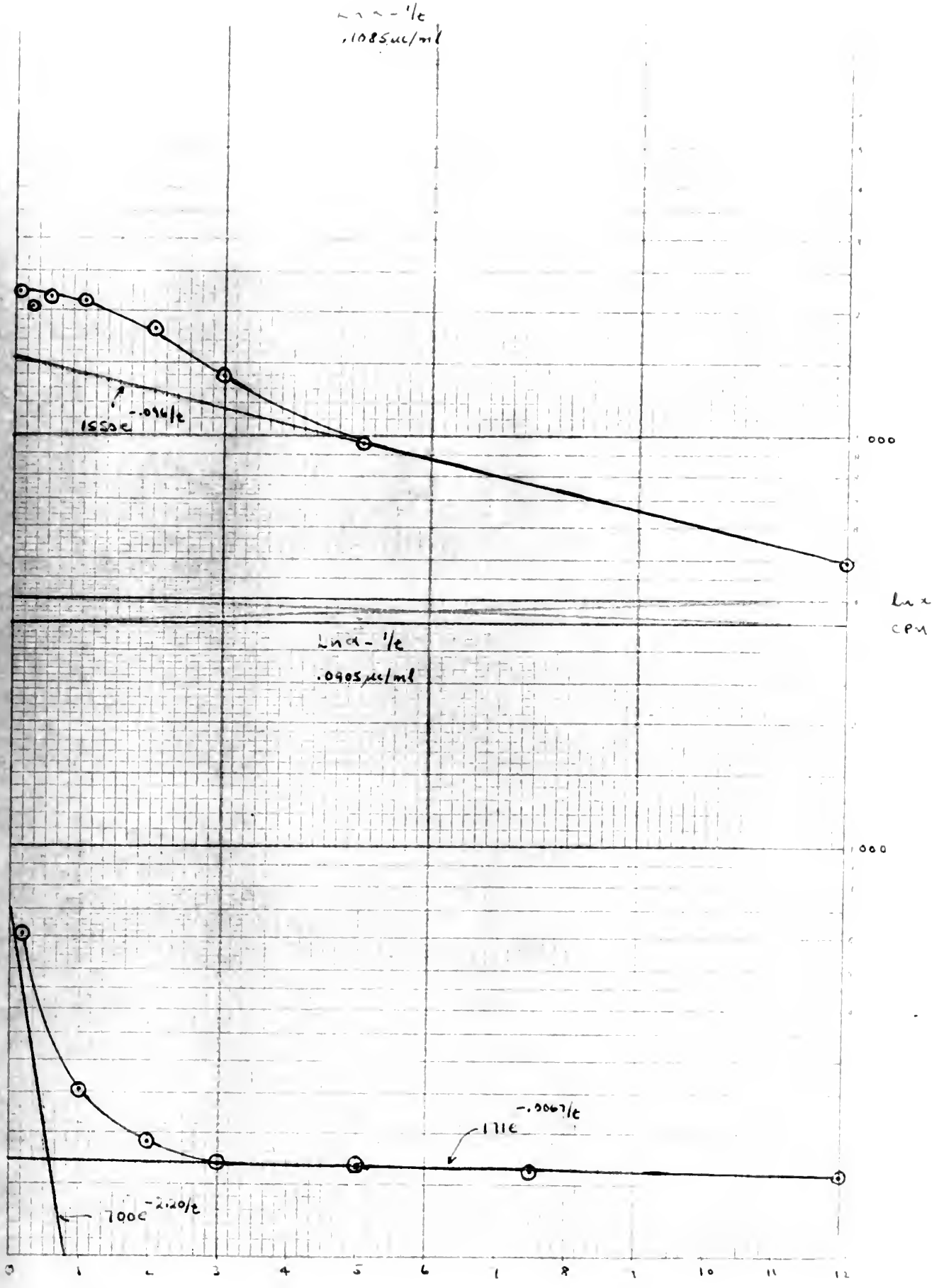
The possibilities outlined above receive some support from the curve plotted on graph I-2 as it is compared to the others in the group. In this plot, the initial adsorption occurs at a higher rate than in the other runs, indicating a surface physically more receptive to the adsorptive. This in turn enables a more rapid build-up of the amount of iodide, with the second phase becoming significant sooner than it might otherwise. In this case the second process takes place less rapidly despite its earlier start, probably because the initial process is still important and taking



place at a significant rate. These two processes must compete for the available adsorptive and hence the effect on the second. In the other runs, the second phase does not become significant as soon, but when it does appear, it does so much more rapidly since the initial adsorption process is more nearly complete.

Surface effects are clearly shown by graphs II and III. The first of these shows the plots of two runs which resulted in marked deviations from the adsorption pattern previously noted. In the upper plot, the planchets followed the normal pattern for the first 30 minutes, and thereafter showed little tendency to adsorb any more iodide. In the light of the postulations of a two-phase process, it would appear here that for some reason the second phase did not proceed at its usual rate, and that the initial adsorption process was only slightly augmented by it. The cause here was unknown, and led to an experiment which produced the lower plot. Here the planchets were prepared in the usual manner, and then were left to soak in a water bath for five days prior to immersion in the active solution. This technique led to the results shown, which indicate that here the first process was grossly inhibited, probably because the available sites were occupied by water molecules. Eventually, after the first 20 minutes, the second phase started and began to build up the surface activity. Since it was possible, by means of surface effects, to inhibit the initial process, it is believed that surface effects could also lead to inhibition of the second process, thus offering a possible explanation of the results shown in the upper plot.

II


Ln c - 1/t
.1085 μc/ml
-.096/t
1550c
000
Ln c
cpm
Ln c - 1/t
.0905 μc/ml
000
-.0067/t
1110
700c
-2.20/t
0
1
2
3
4
5
6
7
8
9
10
11
12
1/t (hr⁻¹)

GRAPH III

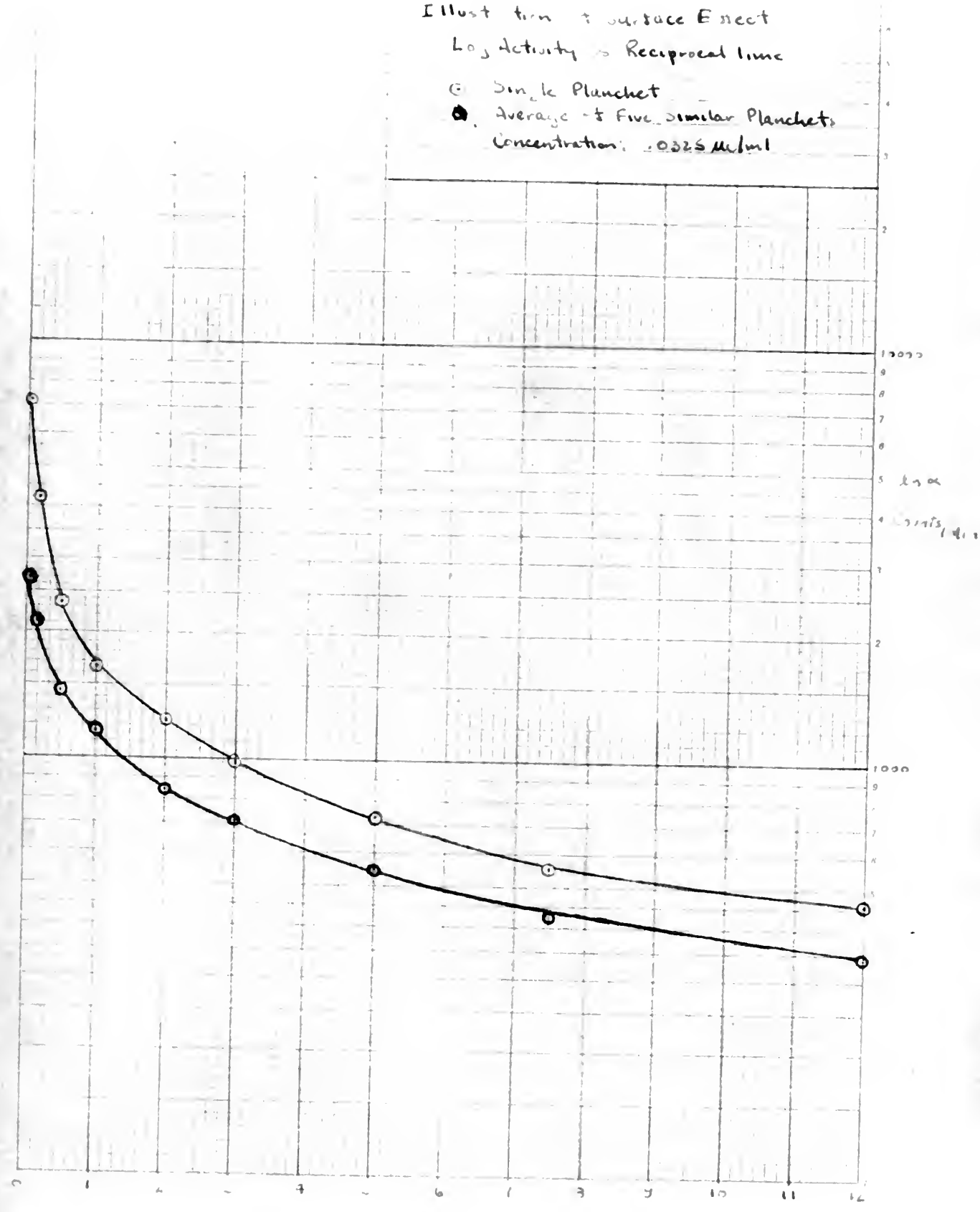
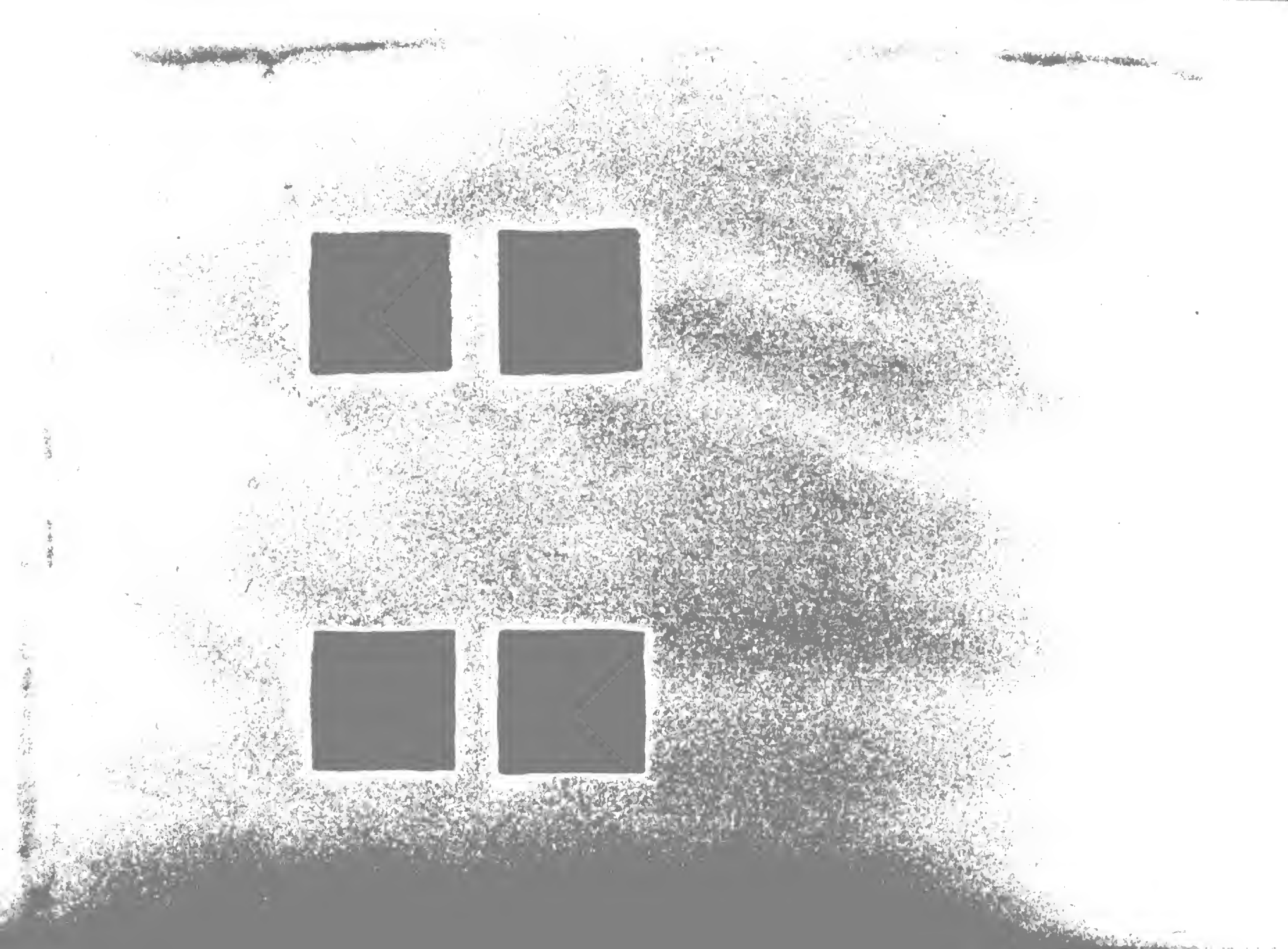


Illustration of Surface Effect
Log Activity vs Reciprocal time
Single Planchet
Average of Five Similar Planchets
Concentration: .0325 Mc/ml
log α
counts / min
1000
1/t (hr⁻¹)



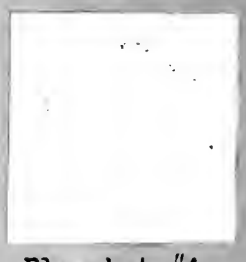
Graph III shows the variation in activity obtained in a particular run using six planchets. One plot represents the average activity on five of the planchets, and the other is a plot of the activity on the remaining planchet. All of the planchets had received identical preparatory treatment and all were immersed in the same solution for the same periods of time. The parallel nature of the curves indicates that surface imperfections may influence the amount of adsorptive that can be collected on the surface without necessarily influencing the general pattern of rate.

A further study of the effects of surface imperfections was made by means of radio-autographs. The planchets from a particular test series were placed on photographic plates and permitted to remain for about 24 hours, after which the plates were developed in the usual manner. The plates were then printed and the results of four of the plates reproduced below.

Planchet #1

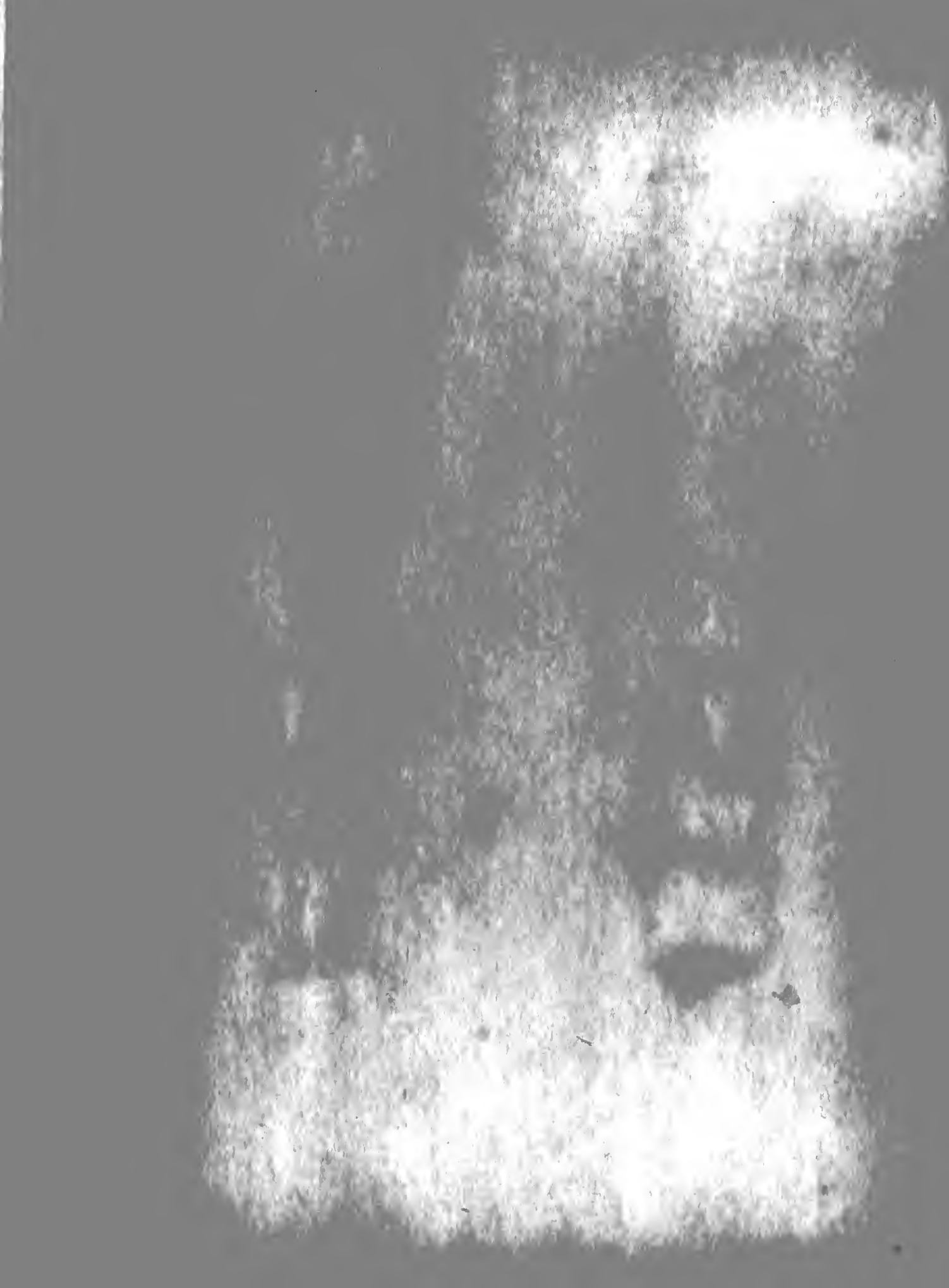
Planchet#2

Planchet #3



Planchet #4

The first planchet was the most active of the group, and clearly in evidence are the numerous hot spots or "active centers" spread over the surface. The reproduction is not so clear as the original plate in this particular feature. The other planchets, in decreasing order of activity,

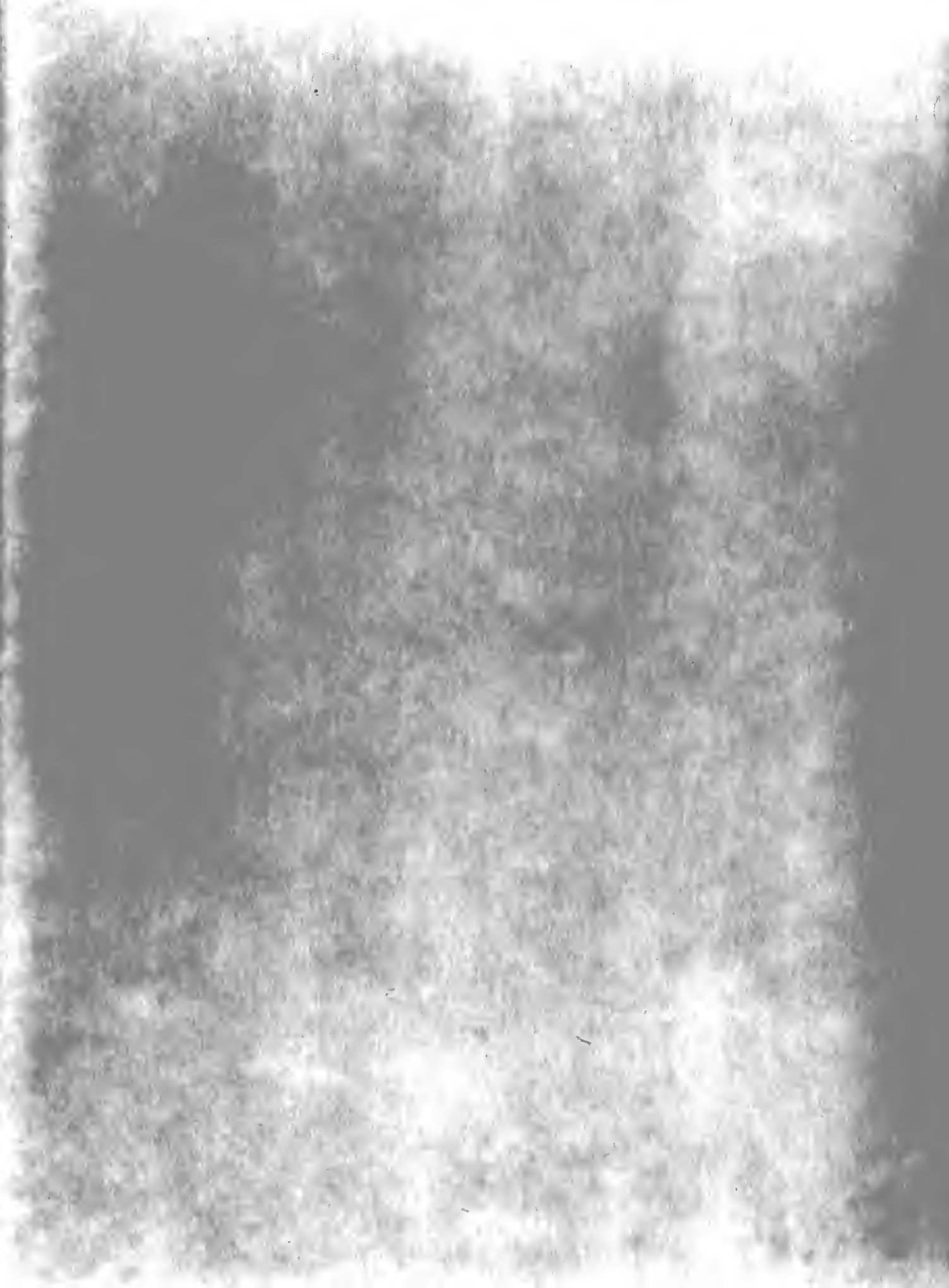


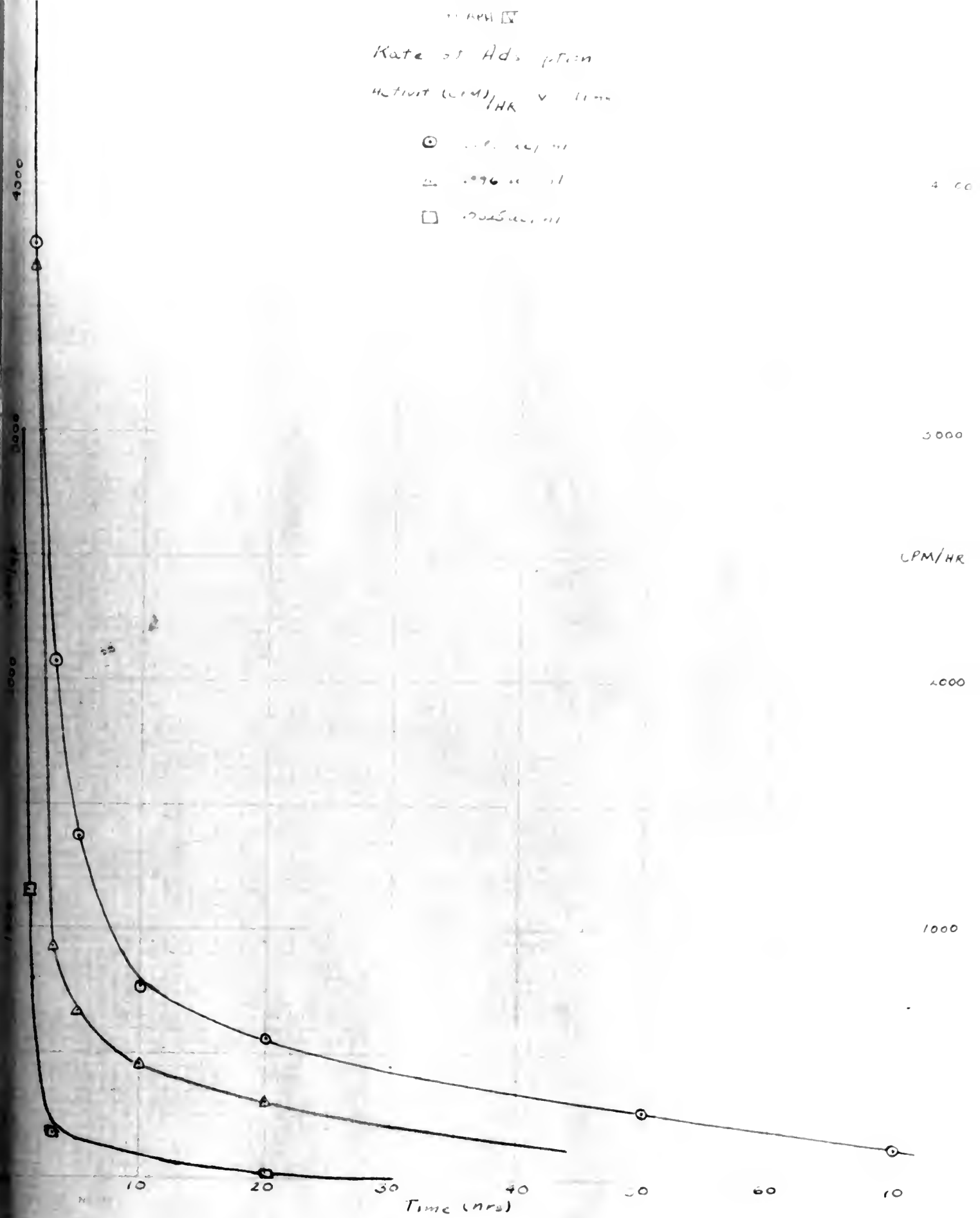
are #3, #4, and #2. On the last planchet, #2, there is a quite uniform appearance to the spread of activity, with almost no noticeable hot spots. Microscopic examination of these same planchets indicated some pitting in the general location of these spots. In general, cracks and fissures were not represented by corresponding concentrations of activity, and this may be due to the bridging of these imperfections by interaction in a lateral sense by the adsorbed molecules.

Graph IV shows the pattern of rate of adsorption. It is clear that the initial rate of adsorption is very high, with an extremely rapid fall-off to low rates within a few hours at most. This follows the pattern which was previously shown in this paper to be among the results of other workers in this field. It also shows a tendency toward equilibrium, in that the rates appear to be falling rapidly toward zero. The low concentration run is most clear in this respect.

Although the empirical relation presented in this paper contains two terms which appear to represent two phases or processes, it may well be that additional terms and hence processes exist. Additional study would be necessary to enlarge upon this possibility. In further studies of this type, it may be advisable to take steps to insure absolute control of concentration in order to isolate this effect from complicating factors. One such step could be to add relatively large amounts of carrier iodine to the solution, using the active iodine in tracer quantities as a measure of adsorption. In this way impurities in the active material would have little effect upon the adsorption process.

The data obtained during this study is of insufficient volume to permit definite conclusions to be made, but certainly there are indic-




Rate of Adsorption
Activity (CPM)/HR vs Time
4000
3000
2000
1000
CPM/HR
10
20
30
40
50
60
Time (hrs)

ations that the overall process may consist of two or more phases, each of which may be affected by the many other complicating factors which may be present. Surfaces are extremely important in adsorption. Careful preparation of the adsorbent may help to minimize surface effects, but it is doubtful if they can be eliminated completely. A factor which may be important, even though only faintly suggested by these data, is pH. The use of an acid bath in preparation of the planchets in this study may have changed the pH slightly in the immediate vicinity of the interface. Indeed, this may have been the mechanism in the case of the water-soaked planchets, since in this case it is doubtful that any acid was left on the planchets after their prolonged water bath. In any event, a study of pH effects would be of considerable interest.

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SERIES #1

Determination of Concentration

Date & Time	Amount of Solution	Counts	Time	CPM	CPM less background	Activity	Activity corrected for Decay
8 Mar 0900	.106ml	8192	56.6 sec	8590	8560	.283μc/ml	.283μc/ml
15 Mar 0900	.105ml	8192	106.0 sec	4640	4602	.153μc/ml	.280 μc/ml

Test Data

Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	CPM Corrected for Decay
8 Mar 0900	1	23.3°c	5 min.	8192	865.5 sec	568	538	538
"	2	"	"	4096	381.0	644	614	614
0930	3	"	"	"	472.7	520	490	490
"	4	"	"	"	562.0	436	406	406
1000	5	"	"	"	427.5	574	544	544
"	6	"	"	"	491.2	500	470	470
8 Mar 1230	1	23.3°c	8 min.	4096	281.5	874	844	844
"	2	"	"	"	259.0	948	918	918
1245	3	"	"	"	367.6	668	638	638
"	4	"	"	"	369.5	665	635	635
1300	5	"	"	"	288.4	864	834	834
"	6	"	"	"	328.0	749	719	719
8 Mar 1310	1	23.3°c	12 min.	4096	235.5	1041	1011	1011
"	2	"	"	"	218.0	1128	1098	1098
1320	3	"	"	"	255.0	962	932	932
"	4	"	"	"	319.0	770	740	740
1335	5	"	"	"	183.5	1340	1310	1310
"	6	"	"	"	253.6	970	940	940
8 Mar 1345	1	23.3°c	20 min.	4096	186.0	1321	1291	1291
"	2	"	"	"	166.0	1480	1450	1450
1355	3	"	"	"	201.8	1220	1190	1190
"	4	"	"	"	227.0	1081	1051	1051
1410	5	"	"	"	156.5	1570	1540	1540
"	6	"	"	"	174.0	1411	1381	1381
8 Mar 1435	1	23.3°c	30 min.	8192	301.0	1632	1602	1602
"	2	"	"	"	265.0	1854	1824	1824
1445	3	"	"	"	314.2	1563	1533	1533
"	4	"	"	"	339.5	1449	1419	1419
1455	5	"	"	"	230.0	2140	2110	2110
"	6	"	"	"	254.0	1936	1906	1906
8 Mar 1510	1	23.3°c	1 hr.	8192	130.2	3775	3745	3745
"	2	"	"	"	159.4	3081	3051	3051
1520	3	"	"	"	155.8	3160	3130	3130
"	4	"	"	"	133.8	3680	3650	3650
1530	5	"	"	"	129.5	3790	3760	3760
"	6	"	"	"	108.5	4530	4500	4500

Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	CPM Corrected for Decay
10 Mar 0930	1	23.0°c	2 hr.	12288	101.0	7295	7257	8640
"	2	"	"	"	107.5	6850	6812	8100
"	3	"	"	"	131.0	5620	5582	6640
"	4	"	"	"	161.8	4555	4517	5370
"	5	"	"	"	109.0	6760	6722	8000
"	6	"	"	"	110.0	6690	6652	7920
12 Mar 1430	1	23.0°c	8 hr.	12288	55.2	13310	13268	19110
"	2	"	"	"	57.1	12890	12848	18520
"	3	"	"	"	65.0	11320	11278	16250
"	4	"	"	"	80.0	9210	9168	13240
"	5	"	"	"	60.0	12288	12246	17690
"	6	"	"	"	59.5	12390	12348	17810
15 Mar 1000	1	23.0°c	75 hr.	16384	37.0	26550	26512	48700
"	2	"	"	"	41.0	23990	23952	44000
"	3	"	"	"	41.5	23650	23612	43500
"	4	"	"	"	67.5	14550	14512	26750
"	5	"	"	"	44.0	22350	22312	41000
"	6	"	"	"	42.0	23400	23362	42900

SERIES #2

Determination of Concentration

Date & Time	Amount of Solution	Counts	Time	CPM	CPM less background	Activity	Activity corrected for Decay
29 Mar 1530	.100ml	2048	44.0 sec	2795	2755	.0960μc/ml	.0960μc/ml
1 Apr 1000	.100ml	2048	56.5 sec	2180	2140	.0747μc/ml	.0952μc/ml

Test Data

Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	CPM Corrected for Decay
29 Mar 1530	1	23.3°c	5min.	4096	124.0 sec	1980	1940	1940
"	2	"	"	"	426.0	577	537	537
"	3	"	"	2048	253.0	486	446	446
"	4	"	"	"	231.0	532	492	492
"	5	"	"	"	208.0	591	551	551
"	6	"	"	"	245.0	502	462	462
30 Mar 0900	1	20.0°c	8 min.	2048	36.0	3420	3280	3490
"	2	"	"	"	108.7	1132	992	1055
"	3	"	"	"	107.0	1150	1010	1074
"	4	"	"	"	121.5	1012	872	929
"	5	"	"	"	108.0	1140	1000	1065
"	6	"	"	"	106.0	1160	1020	1085
30 Mar 0930	1	20.0°c	12 min.	2048	23.2	5300	5160	5500
"	2	"	"	"	79.0	1558	1418	1510
0940	3	"	"	"	89.5	1372	1338	1422
"	4	"	"	"	91.2	1350	1316	1400
"	5	"	"	"	75.8	1621	1587	1690
"	6	"	"	"	62.5	1970	1936	2060
30 Mar 1000	1	21.0°c	20 min.	4096	34.0	7230	7196	7700
"	2	"	"	"	117.0	2100	2066	2215
"	3	"	"	"	118.0	2080	2046	2190
"	4	"	"	"	103.2	2380	2346	2515
"	5	"	"	"	89.4	2750	2716	2905
"	6	"	"	"	75.5	3255	3221	3450
30 Mar 1100	1	21.0°c	30 min.	4096	27.0	9100	9066	9710
"	2	"	"	"	84.5	2910	2876	3080
"	3	"	"	"	84.0	2930	2896	3100
"	4	"	"	"	70.0	3510	3476	3720
"	5	"	"	"	60.5	4060	4026	4310
"	6	"	"	"	57.2	4290	4256	4560
30 Mar 1130	1	21.0°c	1 hr.	8192	41.0	12000	11966	12880
"	2	"	"	"	110.0	4460	4426	4760
"	3	"	"	"	105.0	4680	4646	4998
"	4	"	"	"	92.0	5340	5306	5700
"	5	"	"	"	76.3	6440	6406	6890
"	6	"	"	"	70.2	7000	6966	7495

Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	Decay
30 Mar 1400	1	22.0°c	2 hr.	8192	38.8	12690	12656	13710
"	2	"	"	"	82.8	5940	5906	6400
"	3	"	"	"	77.8	6320	6286	6810
"	4	"	"	"	71.0	6920	6886	7460
"	5	"	"	"	61.8	7950	7916	8580
"	6	"	"	"	57.0	8610	8576	9300
31 Mar 1430	1	22.0°c	7.75 hrs.	8192	32.7	15010	14961	17700
"	2	"	"	"	54.2	9060	9011	10690
"	3	"	"	"	51.0	9640	9591	11380
"	4	"	"	"	52.0	9450	9401	11120
"	5	"	"	"	46.4	10600	10551	12500
"	6	"	"	"	48.7	10100	10051	11900
1 Apr 0950	1	21.0°c	27.0 hr	8192	24.9	19720	19670	24900
"	2	"	"	"	38.0	12920	12870	16290
"	3	"	"	"	34.2	14390	14340	18190
"	4	"	"	"	36.0	13650	13600	17220
"	5	"	"	"	31.0	15870	15810	20050
"	6	"	"	"	35.0	14050	14000	17730

SERIES #3

Determination of Concentration

Date & Time	Amount of Solution	Counts	Time	CPM	CPM less Background	Activity	Activity corrected for Decay
5 Apr 1130	.100ml	4096	251.7 sec	977	937	.0328μc/ml	.0328μc/ml
7 Apr 1300	.100ml	4096	297.0 sec	826	775	.0271μc/ml	.0323μc/ml

Test Data

Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	Decay
5 Apr 1215	1	23.9°c	5 min.	2048	303.0 sec	406	366	366
"	2	"	"	"	341.2	361	321	321
"	3	"	"	"	329.2	374	334	334
"	4	"	"	"	351.5	350	310	310
"	5	"	"	"	246.0	500	460	460
"	6	"	"	"	291.4	422	382	382
5 Apr 1250	1	23.9°c	8 min.	2048	254.0	484	444	444
"	2	"	"	"	289.0	426	386	386
"	3	"	"	"	266.2	461	421	421
"	4	"	"	"	294.0	418	378	378
"	5	"	"	"	206.0	596	556	556
"	6	"	"	"	233.2	527	487	487
5 Apr 1330	1	23.9°c	12 min.	2048	204.2	601	561	561
"	2	"	"	"	229.0	537	497	497
"	3	"	"	"	231.0	532	492	492
"	4	"	"	"	244.4	502	462	462
"	5	"	"	"	158.5	776	736	736
"	6	"	"	"	184.0	669	629	629
5 Apr 1400	1	24.0°c	20 min.	2048	155.8	790	750	750
"	2	"	"	"	174.8	705	665	665
"	3	"	"	"	181.3	678	638	638
"	4	"	"	"	183.7	670	630	630
"	5	"	"	"	120.6	1021	981	981
"	6	"	"	"	139.7	881	841	841
5 Apr 1430	1	24.0°c	30 min.	2048	134.8	915	875	875
"	2	"	"	"	144.0	855	815	815
"	3	"	"	"	143.8	856	816	816
"	4	"	"	"	159.3	771	731	731
"	5	"	"	"	97.2	1268	1228	1228
"	6	"	"	"	122.0	1009	969	969
6 Apr 0915	1	23.2°c	1 hr.	2048	95.0	1296	1251	1350
"	2	"	"	"	121.8	1011	966	1041
"	3	"	"	"	123.3	996	951	1028
"	4	"	"	"	128.4	958	913	985
"	5	"	"	"	78.3	1571	1526	1642
"	6	"	"	"	94.2	1308	1263	1361

Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	CPM Corrected for Decay
6 Apr 1015	1	23.2°c	2 hr.	4096	152.7	1610	1565	1697
"	2	"	"	"	194.0	1267	1222	1325
"	3	"	"	"	198.8	1237	1192	1292
"	4	"	"	"	216.0	1138	1093	1187
"	5	"	"	"	111.0	2215	2170	2355
"	6	"	"	"	139.5	1760	1715	1860
6 Apr 1430	1	24.0°c	6 hr.	4096	105.4	2325	2280	2495
"	2	"	"	"	140.0	1755	1710	1870
"	3	"	"	"	142.5	1723	1678	1835
"	4	"	"	"	163.5	1502	1457	1591
"	5	"	"	"	62.8	3915	3870	4240
"	6	"	"	"	91.0	2700	2655	2905
7 Apr 1145	1	24.0°c	27 hr.	4096	82.0	3000	2949	3475
"	2	"	"	"	97.8	2515	2464	2905
"	3	"	"	"	110.8	2220	2169	2555
"	4	"	"	"	119.2	2060	2009	2365
"	5	"	"	"	39.5	6210	6169	7260
"	6	"	"	"	67.0	3665	3614	4260

SERIES #4

Determination of Concentration

Date & Time	Amount of Solution	Counts	Time	CPM	CPM less Background	Activity
12 Apr 1000	.108ml	4096	72.5 sec	3390	3353	.1085 μc/ml

Test Data

Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	CPM Corrected for Decay
12 Apr 1030	1	24.0°c	5 min.	2048	249.0 sec	494	447	447
"	2	"	"	"	321.3	395	358	358
"	3	"	"	"	256.0	481	444	444
"	4	"	"	"	218.0	564	527	527
"	5	"	"	"	189.7	649	612	612
"	6	"	"	"	211.2	583	546	546
12 Apr 1100	1	24.0°c	12 min.	4096	289.0	850	813	813
"	2	"	"	"	330.0	744	707	707
"	3	"	"	"	266.0	924	887	887
"	4	"	"	"	222.5	1102	1065	1065
"	5	"	"	"	200.8	1221	1184	1184
"	6	"	"	"	217.2	1130	1093	1093
12 Apr 1130	1	24.0°c	20 min.	4096	227.0	1082	1045	1045
"	2	"	"	"	236.0	999	962	962
"	3	"	"	"	176.8	1390	1333	1333
"	4	"	"	"	155.7	1580	1543	1543
"	5	"	"	"	125.0	1968	1931	1931
"	6	"	"	"	154.8	1589	1552	1552
12 Apr 1200	1	24.0°c	30 min.	4096	173.3	1418	1381	1381
"	2	"	"	"	165.5	1485	1448	1448
"	3	"	"	"	129.0	1905	1868	1868
"	4	"	"	"	117.0	2100	2063	2063
"	5	"	"	"	107.2	2285	2248	2248
"	6	"	"	"	132.0	1861	1824	1824
12 Apr 1230	1	24.0°c	1 hr.	4096	139.3	1762	1725	1725
"	2	"	"	"	119.2	2060	2023	2023
"	3	"	"	"	121.2	2025	1988	1988
"	4	"	"	"	101.0	2435	2398	2398
"	5	"	"	"	98.0	2510	2473	2473
"	6	"	"	"	117.8	2085	2048	2048
12 Apr 1345	1	24.0°c	2 hr.	4096	124.8	1970	1933	1933
"	2	"	"	"	105.0	2340	2303	2303
"	3	"	"	"	126.3	1942	1905	1905
"	4	"	"	"	98.2	2500	2463	2463
"	5	"	"	"	98.5	2495	2458	2458
"	6	"	"	"	126.0	1950	1913	1913

Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	CPM Corrected for Decay
12 Apr 1545	1	24.5°c	4 hr.	4096	121.2	2025	1988	1988
"	2	"	"	"	107.5	2285	2248	2248
"	3	"	"	"	141.0	1742	1705	1705
"	4	"	"	"	105.0	2340	2303	2303
"	5	"	"	"	109.0	2255	2218	2218
"	6	"	"	"	137.2	1790	1753	1753
13 Apr 0730	1	23.0°c	19.5 hr	4096	105.2	2325	2279	2455
"	2	"	"	"	101.7	2420	2374	2560
"	3	"	"	"	132.3	1855	1809	1948
"	4	"	"	"	108.7	2260	2214	2385
"	5	"	"	"	115.8	2120	2074	2235
"	6	"	"	"	145.0	1692	1646	1772

SERIES #5

Determination of Concentration

Date & Time	Amount of Solution	Counts	Time	CPM	CPM less Background	Activity	Activity Corrected for Decay
13 Apr 0800	.108ml	4096	86.2 sec	2845	2799	.0905μc/ml	.0905μc/ml
13 Apr 1600	.108ml	4096	89.0 sec	2760	2714	.0879μc/ml	.0905μc/ml

Test Data

Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	CPM Corrected for Decay
13 Apr 0800	1	22.0°c	5 min.	2048	343.3 sec	358	312	312
"	2	"	"	1024	706.0	87	41	41
"	3	"	"	"	173.7	354	308	308
"	4	"	"	"	354.6	173	127	127
"	5	"	"	"	470.0	131	85	85
"	6	"	"	"	574.0	107	61	61
13 Apr 0815	1	22.0°c	10 min.	1024	184.7	332	286	286
"	2	"	"	"	434.0	142	96	96
"	3	"	"	"	234.2	262	216	216
"	4	"	"	"	262.4	234	188	188
"	5	"	"	"	302.0	204	158	158
"	6	"	"	"	528.0	116	70	70
13 Apr 0930	1	22.0°c	20 min.	1024	452.4	136	90	90
"	2	"	"	"	496.0	124	78	78
"	3	"	"	"	179.0	343	297	297
"	4	"	"	"	220.0	279	233	233
"	5	"	"	"	271.0	227	181	181
"	6	"	"	"	360.0	171	125	125
13 Apr 1020	1	22.0°c	1 hr.	1024	334.0	184	138	138
"	2	"	"	"	308.0	200	154	154
"	3	"	"	"	142.3	431	385	385
"	4	"	"	"	159.8	385	339	339
"	5	"	"	"	181.7	338	292	292
"	6	"	"	"	236.5	260	214	214
13 Apr 1600	1	23.0°c	6 hr.	1024	102.5	600	554	570
"	2	"	"	"	104.0	591	545	561
"	3	"	"	"	65.2	941	895	921
"	4	"	"	"	86.7	709	663	682
"	5	"	"	"	109.0	564	518	534
"	6	"	"	"	121.0	507	461	475

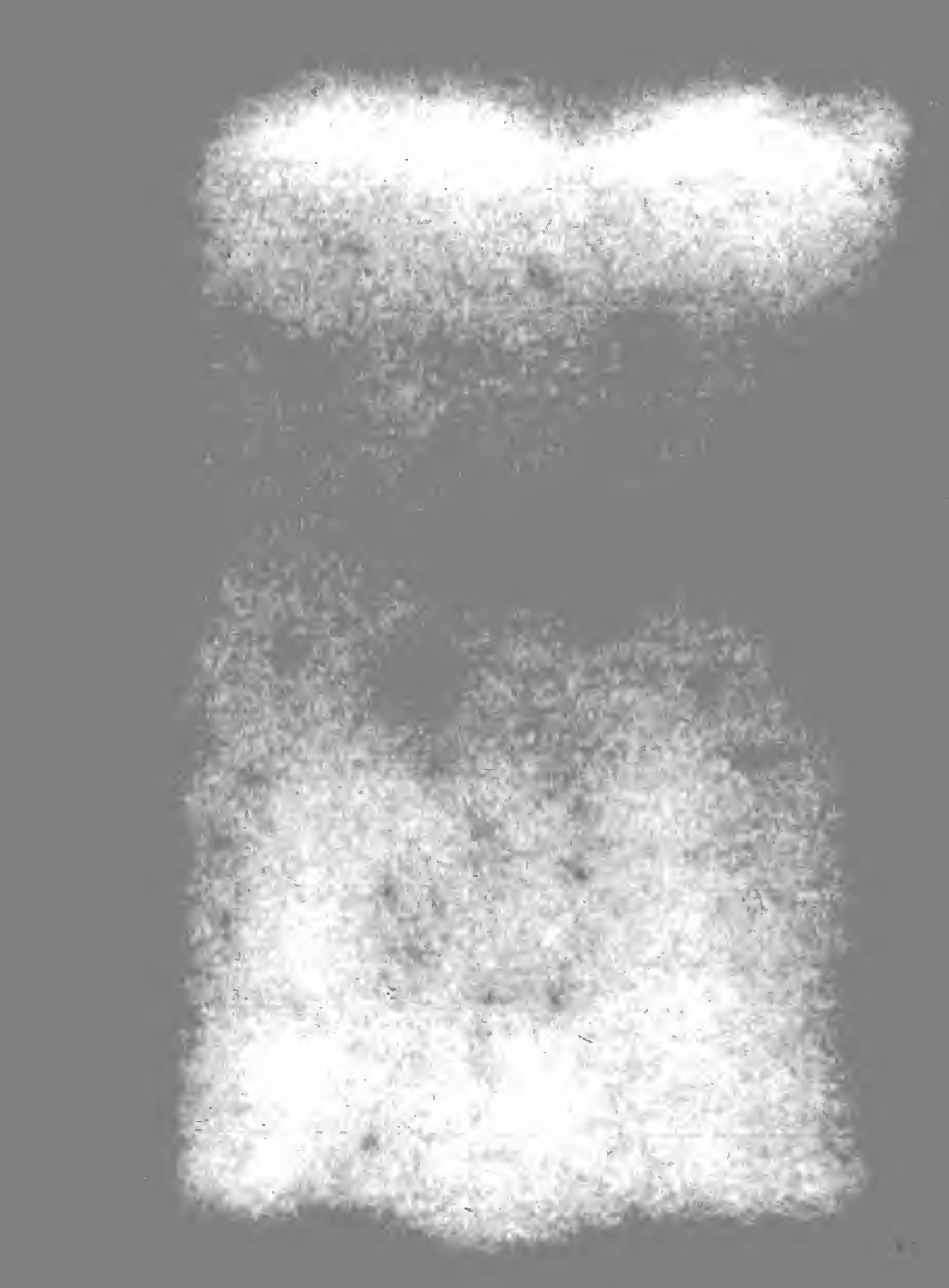
SERIES #6

Determination of Concentration

Date & Time	Amount of Solution	Counts	Time	CPM	CPM less background	Activity	Activity Corrected For Decay
13 Apr 1600	.108ml	4096	88.6 sec	2766	2720	.0880µc/ml	.0880µc/ml
15 Apr 1300	.106ml	4096	108.0 sec	2275	2236	.0736µc/ml	.0876µc/ml

Test Data

Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	CPM Corrected for Decay
13 Apr 1600	1	24.0°c	5 min.	1024	126.5sec	486	440	440
"	2	"	"	"	107.0	575	529	529
"	3	"	"	"	96.3	639	593	593
"	4	"	"	"	129.0	476	430	430
"	5	"	"	"	117.4	524	478	478
"	6	"	"	"	126.8	486	440	440
13 Apr 1615	1	24.0°c	10 min.	1024	85.5	720	674	674
"	2	"	"	"	74.0	831	785	785
"	3	"	"	"	73.0	842	796	796
"	4	"	"	"	81.8	752	706	706
"	5	"	"	"	80.2	766	720	720
"	6	"	"	"	83.4	739	693	693
14 Apr 0900	1	23.0°c	20 min.	1024	68.0	904	848	910
"	2	"	"	"	60.2	1020	974	1031
"	3	"	"	"	58.3	1055	1009	1070
"	4	"	"	"	76.7	801	755	800
"	5	"	"	"	68.3	900	854	905
"	6	"	"	"	72.5	848	802	850
14 Apr 0920	1	23.0°c	30 min.	2048	126.0	976	930	986
"	2	"	"	"	108.0	1140	1094	1160
"	3	"	"	"	112.8	1091	1045	1108
"	4	"	"	"	133.4	921	875	927
"	5	"	"	"	123.5	996	950	1007
"	6	"	"	"	133.2	924	878	931
14 Apr 1000	1	23.0°c	1 hr.	2048	107.0	1150	1104	1181
"	2	"	"	"	95.0	1295	1249	1337
"	3	"	"	"	97.8	1259	1213	1300
"	4	"	"	"	114.3	1076	1030	1101
"	5	"	"	"	105.7	1165	1119	1198
"	6	"	"	"	116.0	1061	1015	1087
14 Apr 1100	1	23.0°c	2 hr.	2048	95.0	1295	1249	1337
"	2	"	"	"	80.0	1540	1494	1600
"	3	"	"	"	87.9	1400	1354	1450
"	4	"	"	"	95.0	1295	1249	1337
"	5	"	"	"	89.3	1380	1334	1429
"	6	"	"	"	98.2	1252	1206	1290



Date & Time	Planchet #	Temp.	Total Immersion Time	Counts	Time	CPM	CPM Corrected for Background	CPM Corrected for Decay
14 Apr 1530	1	24.0°c	6.5 hr.	2048	78.6	1565	1519	1652
"	2	"	"	"	60.0	2048	2002	2225
"	3	"	"	"	76.2	1612	1566	1708
"	4	"	"	"	84.0	1465	1419	1545
"	5	"	"	"	69.5	1770	1724	1680
"	6	"	"	"	58.5	2100	2054	2240
15 Apr 1230	1	23.5°c	27.25 hr.	2048	63.3	1945	1906	2240
"	2	"	"	"	49.8	2465	2426	2850
"	3	"	"	"	63.0	1954	1915	2245
"	4	"	"	"	64.0	1921	1882	2215
"	5	"	"	"	53.0	2320	2281	2680
"	6	"	"	"	26.0	4730	4691	5500

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